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INFLUENCE OF OXIDATION PARAMETERS ON ROUGHNESS AT THE SI-SIO₂ I--ETC(U)
JAN 80 M HENZLER, P HAHN DA-ERO-78-8-011
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Influence of Oxidation Parameters on
Roughness at the Si-SiO₂ Interface

Final Technical Report

by

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January 80

EUROPEAN RESEARCH OFFICE
United States Army
London, England

Grant Number DA-ERO 78-G-011
M. Henzler, Technical University, Hannover

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80 5 27 065

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S u m m a r y

The roughness at the interface Si-SiO₂ has been determined on an atomic scale after removal of the oxide by LEED (Low Energy Electron Diffraction). The energy dependant broadening of the diffracted electron beams yields the average size of step free terraces. Silicon (111) samples have been oxidized under various conditions concerning atmosphere (dry and wet oxygen), temperature (800^o C and 1000^o C), time, pretreatment and posttreatment. The oxidation process produces a roughness, which may be decreased by low oxidation rates and appropriate annealing in non-oxidizing atmosphere. The novel technique of evaluation for the first time shows systematically, how oxidation parameters determine the roughness at the interface, which again is important for the performance of MOS-devices.

Keywords: Si-SiO₂ interface
 LEED (Low Energy Electron Diffraction)
 Roughness determination
 Structural properties
 Silicon surface

I. Introduction

The quality of MOS-devices depends among other parameters strongly on the quality of the interface between silicon and silicon-dioxide. Whereas the chemical composition (like segregation of alkali ions) and electrical properties (interface states, interface charge, scattering of electrons at the interface) have been studied for a long time, the structural properties had been neglected due to lack of appropriate measuring techniques. Only recently several new techniques have been applied. Depth profiling by ion bombardment and monitoring of the varying chemical composition of the surface during removal of the oxide provides some information on a "transition layer". The assumption, however, is required that the ion beam does not produce a roughness of the same order of magnitude. Transmission electron microscopy of cross sections of MOS structures shows directly the profile of the interface. A quantitative interpretation is still difficult.

A novel technique for studying the interface uses electron diffraction (LEED). If the surface of a single crystal shows different levels due to atomic steps, the diffracted electron beam is modified by interference of the electrons diffracted at adjacent terraces. By varying the energy and therefore also the wave length of the electrons this interference changes periodically between constructive and destructive interference. Therefore the spot shape changes periodically with increasing energy. From the period the step height is derived with high precision ($< 1\%$ of step height), from the spot shape the distribution of terraces with respect to orientation, width and regularity is obtained (for details see references 1 - 4).

This method may be applied also to study the interface of Si and SiO_2 . After removal of the oxide the roughness of the interface is seen directly due to the broadening of the

diffraction spots, which changes periodically with electron energy. Whereas the Annual Technical Report (March 79) has presented the first systematic results, the present Final Report shows, how the oxidation time, temperature and treatments before and after oxidation influences the roughness at the interface as measured after removal of the oxide. The theoretical aspects of the interpretation of the LEED-pattern have been reviewed and are part of this report (already published in Adv. in Sol. State Physics XIX, p 193, J. Treusch (ed.), Vieweg, Braunschweig 1979).

II. Experimental

For the experiments a UHV-system has been set up with a four-grid-LEED system (Physical Electronics). The LEED spots at the phosphoric screen have been profiled with a closed TV-system (Hamamatsu). A sample transfer system enabled a quick exchange of samples. The details have been described in the Annual Report (March 79). In the meantime two improvements have been added: an automated recording and evaluation of the LEED spot profile and an extension of oxide thickness measurements to very low thickness with the help of Auger Electron Spectroscopy.

a) On-line LEED spot profiling.

The LEED spots at the phosphoric screen have been profiled with a closed TV-system (Hamamatsu). The schematic configuration is seen in fig. 1. The system may be operated with 256 up to 1024 lines per image. One vertical column of spots of the image at an arbitrary horizontal position is digitalized and shown as an intensity profile within the screen of the monitor. By rotation of the camera an arbitrary cross section of a spot may be chosen.

The intensity profile is first recorded by a transient recorder which is able to store 2048 numbers with 8 bit resolution. By use of an on-line computer (PDP 11) the data may be recorded directly by an xy-recorder or processed for any kind of evaluation.

The software (in FORTRAN) enables a wide variety of procedures. In the usual procedure the program first determines the distance of the 00-spot from the 10 spot on the screen, since the half width of the spots has to be calculated relative to this distance (see Annual Report, March 79). For determination of the half width the following data are given to the computer: energy of electrons, number of cycles for accumulation, time of integration for each cycle. The computer provides the optimum voltage for focussing of the electron beam, which (so far) has to be done manually. Then the computer collects and accumulates the intensity profiles, selects a field of 200 points each on both sides of the maximum spot, determines and subtracts the background and calculates the half width relative to the next spot distance. After manual change of the electron energy or sample position the procedure is repeated.

The FORTRAN program and some of the macro subroutines are given in the appendix.

The determination of edge atom densities from relative half width is already given in the Annual Report (march 79) and in the included review paper.

b) Oxide thickness determination for very thin oxides

To extend the measurements of roughness to interfaces at very thin oxide layers, an alternative method to the Tolanski method (see Annual Report, March 79) has been applied which is limited to thickness $d > 15$ nm. With Auger

Electron Spectroscopy the binding energy of silicon atoms may be measured. Since in oxide this binding energy is shifted by a few electron volts (chemical shift), a high resolution electron spectrometer can distinguish between silicon atoms in oxide and in bulk silicon. C.C. Chang and D.M. Boulin (5) have calibrated this method by a determination of the exponential decay of electron escape probability for different distances of the originating silicon atom from the surface. At an energy of 1615 eV they found an escape depth of 3.1 ± 0.9 nm in SiO_2 . The method is suited up to oxide thickness of 12 nm.

A commercial cylindrical Auger Electron Spectrometer (Physical Electronics) with an energy resolution of 0.3%, lateral resolution of 5 μm and an coaxial integral gun has been used. The spectra have been recorded with a 2 volt peak-to-peak modulation and an incident electron beam of 5 - 10 μA at 6.6 keV. In fig. 2 it is seen that the clean (a) and heavily oxidized surface (c) show two easily distinguishable recordings. From the intensity ratio at thin oxides (b) the oxide thickness is derived.

III Influence of procedure on the results

a) Cleanliness of samples after removal of oxide

To find out the state of the surface after removal of the oxide, Auger Electron Spectroscopy has been applied to several samples, treated as for the LEED experiments.

The samples checked were oxidized with various oxidizing parameters. One sample was oxidized by Siemens AG, München, within their commercial standards. Some AES spectra are shown in fig. 3. Besides silicon (92 eV) only oxygen

(503 eV) and carbon (272 eV) is found. No other contamination was detectable. The peak-to-peak signals are representative for the surface concentrations of the respective elements. The ratio of the signals of Si to C and O was about 100:5:5 for the commercial sample and nearly identical (100:7:7) for the own samples. According to the calibrations as reported by Kasupke (6) and Klimesch (7) the coverage of carbon and oxygen is about 0.3. It is obvious that the surface is not clean on an atomic scale. On the other hand this amount of contamination just can produce some background of the LEED pattern, it, however, can not influence the oscillation of the half width of the LEED spots. Therefore the conclusion is supported, that the observed oscillations are due to the step structure of the silicon itself.

After many cycles of sample exchange the sample holder (which is a single crystal of silicon itself) showed a much higher carbon and oxygen coverage, although it had been etched each time in HF together with the samples. Therefore the sample holder was contaminated with hydrocarbons and other compounds (containing hydrogen, carbon and oxygen), which were not removed by etching (see fig. 3.b). This contamination increased the background in the LEED pattern and deteriorated the LEED spots. Oxidizing the holder and etching together with the samples restored a bright LEED pattern.

b) Influence of treating in nitrogen, air and methanol

Since the samples between etching in HF and observing in UHV have to be handled in liquids and atmospheres, it is necessary to check, if those parts of the procedure have any influence on the result. For that purpose samples have been heated up to 1100° C in UHV and checked for surface

roughness in situ. The pattern showed the usual 7x7-superstructure and no oscillation of the halfwidths of any spot. Therefore at the beginning the samples were free of steps within the limits of the experiments. Such a sample was treated like a sample etched in HF, to see if any roughness occurs. Starting in UHV, the atmospheric impacts were gradually increased: 1) A short shift into the lock at 5×10^{-6} mbar and back to UHV. 2) Exposure to nitrogen within the lock up to 10^{-1} mbar. 3) Exposure to 1 bar of nitrogen within the lock. 4) Exposure to air for 15 sec in the lock. 5) Dipping into methanol after exposure to air and transfer back to vacuum with a droplet of methanol. After each step the sample was transferred back to UHV, checked for roughness and annealed at 1100° C before the next step.

As a result each of the five steps given above increases the background more and more from step one to step five and decreases the intensities within the spots. Up to step 4 the spots are still visible. They do not show any broadening compared to the well annealed structure. Therefore no atomic steps are produced by the parts of the procedure described above.

As a next step the well annealed sample has been brought from the methanol dip into HF and then treated as usual (transfer under methanol into UHV). The result is shown in fig. 4. The lower line gives the instrumental limit. The crosses are the experimental points and the upper line is a calculated curve for a half width of 2.7%, corresponding to an edge atom density of 3.8%. Seven runs yielded similar values. Somewhat lower values have been obtained, when the lock was filled back to atmospheric pressure by nitrogen instead of air.

This residual roughness is considerably lower than that observed for an oxidized sample. An example is shown in

fig. 5. Whereas the oxidized surface (30 sec at 1000°C in dry oxygen) showed a half width of about 12%, (crosses x x x, upper line), the same sample yields after annealing to 1000°C in ultra high vacuum a smooth curve without oscillations (circles, lower line). After bringing back to air and an etch in HF as described above a small half width of 3% is observed (crosses + + + and middle line).

These experiments show, that the procedures have a low influence on the observed roughness. It is therefore concluded, that the reported results are due to the interface and not due to handling after etching.

c) Influence of multiple etching, of stains and of storage in air

If the roughness as measured after oxidation is not produced by the etching procedures, it should not be affected by repetition of the etching. For that proof, an oxidized sample (16 h at 1000°C in dry oxygen) has been measured. Fig. 6 shows the half width immediately after removal of the oxide, and the same sample after transfer out of the system into HF and back to the system and after a second repetition of the procedure of etching in HF. It is seen that the half width is always $11.7\% \pm 0.5\%$. The results show that repeated etching does not influence the measured half width of an oxidized sample. After annealing of the sample and repeating the etching procedure again a very low half width of 3% (circles in fig. 6) has been found.

Sometimes due to a less careful handling of the samples before oxidation the oxides showed stains and a local variation of the oxide thickness. Those samples showed both a high average half width and a high variation of it over the sample (see fig. 7, upper curve). Those samples

have been disregarded for further evaluations.

A sample, well annealed in UHV has been stored in air over night. Due to that light oxidation (the thickness has not been determined) a small roughness has been observed (see Fig. 7, lower curve, relative half width $5,1 \pm 0,7\%$). A thin oxide therefore produces only a low roughness.

d) Variation of the etching procedure

In all experiments the samples were mounted on the sample holder (out of silicon), dipped together with the sample into methanol and then with a coverage of methanol transferred into the etchant HF. It has been checked, if the coverage with methanol during transfer into the etchant is important.

Therefore for several identically oxidized pairs of samples one sample has been etched with a methanol coverage, the other without it. The results for seven different oxidation parameters are shown in fig. 8. Always the circles refer to etching without methanol and the crosses to etching with it. It is clearly seen that the runs without methanol show an appreciable additional roughening due to the etchant. This is especially drastic for the samples six and seven, which have been annealed in nitrogen at 750°C after oxidation. Obviously the reactivity of the etchant is drastically altered by the absence of methanol. Therefore all following results were obtained with dipping into methanol before etching in HF. In this way an additional roughening due to the etching procedure has been avoided.

IV Results

a) Oxidation at 800° C

Two Si(111)-samples had been annealed each time in UHV at 1000° C to the ideal 7x7 superstructure prior to oxidation. After transfer into the quartz tube they were oxidized in dry oxygen at 800° C for various times (1 min to 20 hours). The oxide thickness was determined (as described in section II) by a Tclanski microscope or by Auger Electron Spectroscopy. The half width of the OO-beam in the LEED pattern was measured for several electron energies between 30 and 110 eV. All reported values are averages of many spots at the sample. The error bars are the standard deviations from the average.

Fig. 9 shows the relative half width (reduced by the instrumental broadening) and the calculated step atom density for many different oxide thicknesses. The crosses are results from one sample after repeated oxidations (with annealing before each oxidation), the circles for another sample. The main result is, that for low oxide thickness (< 20 nm) the edge atom density is appreciable higher (25%) than for higher thickness (only 15%). Obviously a change in the oxidation process occurs at about 20 nm. In fig. 9 also results with wet oxygen are reported (symbol W1 and W2). Sample 1 was well annealed before oxidation and sample 2 was oxidized and etched in HF (without transfer into UHV) before final oxidation. Both samples show an appreciable higher roughness.

The results clearly demonstrate, that both the oxide thickness and the atmosphere have a strong influence on the roughness at the interface between oxide and silicon.

b) Oxidation at 1000° C

The results already reported in the Annual Report (march 1979) for oxidation at 1000° C in dry oxygen have been repeated, confirmed and extended down to thicknesses of about 23 nm. For even thinner oxides the oxidation time would have been too short. The results are shown in fig.10. Considering the error bars and the extended range the roughness may be described as independent of oxide thickness. This roughness is somewhat lower than that observed after oxidation at 800° C in the same thickness range. One sample (W3) has been oxidized in wet oxygen at 1000° C. Again the roughness is considerably higher (fig. 10, symbols W3).

c) Variation of surface condition before oxidation

The so far reported results have been obtained from samples, which were step free before oxidation due to annealing in ultra high vacuum. To find out, if the condition before oxidation influences the result, four different starting conditions have been checked:

- 1) Factory polished (by Wacker Chemitronic, Burghausen).
- 2) Chemically etched with CP6.
- 3) Oxidized at 1000° C and oxide removed by HF.
- 4) Step free due to annealing in UHV.

All samples were cleaned ultrasonically and oxidized simultaneously at 1000° C for about 2 hours. The half width was measured for two different electron energies and averaged for many positions on the sample. The average values and their standard deviations are shown in fig. 11. It is seen, that an improvement is obtained by chemical etching, an even better improvement by a first oxidation or an annealing in UHV. To confirm the drastic difference of the first and the third starting condition, such samples

have been oxidized simultaneously in a second run. The results are shown at the right edge of fig. 11. Even at a somewhat higher level of roughness the drastic improvement due to the first oxidation again is evident.

d) Posttreatment after oxidation

All so far reported results have been obtained from samples which were measured immediately after oxidation. To see, if a non-oxidizing posttreatment would vary the results, some of the samples out of the second run in section c) (fig. 11) have been treated after oxidation in nitrogen at 750°C for 5 hours. All samples used for fig. 12 were oxidized simultaneously, some only factory polished, some with prior oxidation and removal of oxide. For both groups the posttreatment in nitrogen yields an appreciable decrease of roughness. The smoothing effect of the posttreatment is also seen in the much smaller scattering of the roughness for different positions on the sample.

V Discussion

a) Reliability of the method

The interface between Si and SiO_2 cannot be studied in situ. The oxide has to be removed. It is a question if the roughness found after removal of the oxide is the same as present at the interface. The experiments of section III show, that all parts of the procedure except the etching in HF do not affect the measured roughness. Also multiple etching did not vary the result. Since it is not possible

to check an etching in HF of an absolutely oxide free silicon surface (any handling at atmospheric pressure produces some minor oxidation), it is only very likely although not proven to the final point, that the procedure does not alter the roughness at the interface. Some results support the conclusion, that the surface under investigation is the wanted interface with respect to roughness: 1) after minimum oxidation (quick handling in atmospheric pressure, no oxidation on purpose) a minimum roughness is observed. 2) The results are reproducible. 3) The measured roughness depends on all oxidation parameters. 4) The measured increase in roughness for low oxide thickness (see fig. 9) coincides with the thickness range, where the growth kinetics is controlled by reactivity at the interface. The lower roughness is only found for oxides, where the growth is diffusion controlled. The measured roughness therefore supports models of growth kinetics (see also the following section b).

Since so far it is not possible to measure surface roughness on an atomic scale with any other technique, a quantitative check of the reliability of the method cannot be given. The qualitative checks, however, make evident, that the measured roughness is highly relevant for the status of the interface before removal of the oxide.

b) The effect of oxidation parameters

The results demonstrate that the interface show an appreciable number of edge atoms. The average terrace width varies from 6 to 40 atomic distances. After annealing at 1000° C in ultra high vacuum the average terrace width is beyond the limits of the method (more than 100 atomic distances). Therefore the measured roughness is produced during oxidation. The roughness strongly depends on the oxidation parameters. The oxidation process occurs after

diffusion of oxygen through the oxide. Due to random fluctuations the attack may be different at different spots of the surface, so that the interface may switch between several layers of the silicon sample. Therefore random attack in reactivity of oxygen is the roughening factor at the interface, which should pile up to more and more roughness with growing oxide thickness, if there would be no smoothing effect. In the case, there is no or not sufficient supply of oxygen at the interface, a smoothing effect has to occur due to diffusion of oxygen at the interface. Similar to the free silicon surface also at the interface a smooth, step free surface is energetically favored compared to a stepped surface. Any diffusion of oxygen at the interface therefore has to smooth the silicon surface under the oxide, until a step free surface is obtained. Since no step free interface has been found, either equilibrium has not been obtained or equilibrium implies a fairly high number of atomic steps due to the entropy contribution to the Helmholtz energy of equilibrium. By using both the roughening and the smoothing effect the results of section IV may be described congruently: In fig. 9 a decrease of roughness for oxide thickness more than 20 nm is observed. If the oxide thickness is plotted versus the oxidation time a linear increase is observed up to that thickness followed by a square root dependance. This result is in agreement with Deal and Grove (8), who interpret the linear increase as reaction controlled and the square root increase as diffusion controlled. If diffusion through the oxide limits the supply a smoothing at the interface should occur. This is observed with the decrease of the roughness. The further decrease of roughness for oxidation at 1000° C (fig. 10) obviously is due to a higher diffusion at the interface.

A strong support of the smoothing at the interface is given by the result, that any annealing in non-oxidizing

atmosphere yields an additional substantial decrease of roughness (fig. 12). On the other hand oxidation in wet oxygen increases the oxidation rate by more than one order of magnitude. Therefore again the reactivity and therefore the roughening effect is increased compared to the smoothing effect. It is a straight forward consequence, that the roughness has to be higher, as found experimentally (figs. 9 and 10).

The dependence on starting condition (fig. 11) yields an additional important factor. Both structural and chemical differences may be the reason. It is, however, difficult to maintain structural differences (like crystallographic distortions by mechanical polishing) at 800°C or 1000°C , since surface diffusion and recrystallisation should restore an ideal surface very quickly. Chemical differences, especially metal or carbon contamination, should vary the electronic properties and therefore the chemical reactivity at the interface substantially, as found in detail on the free, clean silicon surface. The decreasing roughness in fig. 11 may therefore interpreted as increasing cleanliness to start with. Whereas a factory polished sample should show the highest contamination (due to storage in between), even the chemical etch shows more contamination (as proved with AES). Cleaning in ultra high vacuum and a first oxidation obviously yield the same, sufficient cleaning for the present purpose.

The present results therefore for the first time yield direct information, how oxidation parameters influence the roughness at the interface. It is seen, how the treatment has to be varied to yield a low roughness.

c) Comparison with other techniques

Due to the high importance of the Si-SiO₂ interface for the performance of MOS-devices a wide variety of methods and techniques have been developed to study both the structural and the chemical and the electronic properties of the interface. Most of the recent results are found in conference proceedings (9 , 10 , 11). Those methods like Rutherford scattering (12), AES (13,14), XPS (15,16) and UPS (17), Low energy ion scattering (18), contact angle of a liquid (19), TEM (10, 11, 20, 21) and electronic mobility in the space charge layer indicate the presence of a transition layer the thickness of which is estimated to 0.3 - 5 nm, depending on the methods. Some methods cannot distinguish between a gradual change of the chemical composition within the transition layer and an undulation of the interface with an asperity height as thickness of the transition layer.

The present results give information strictly on structured properties. Especially the average terrace width or the edge atom density are given, which without further assumption may not be used to calculate a thickness of a transition layer. The information is on an atomic scale and somewhat different in nature compared to other techniques, which measure mostly the average chemical or electronic structure after production or removal of an oxide layer (or part of it).

Nevertheless those methods provide some information on structural properties, especially when the oxide layer is removed by ion milling in many small steps (depth profiling by ion bombardment). Measurements of Johannessen, Spicer and Strausser (13) indicate, that during ion milling a constant transition layers for oxides of 10 to 200 nm thickness. For very thin oxides Wagner and Wilmsen (14) found an increase of the transition layer from 1 to 2.5 nm for oxides from 2 to 4.5 nm. Both results

are compatible with our results, although the kind of information is different. The most severe assumption used is, that the ion bombardment itself does not produce an appreciable transition layer by intermixing of the top layers and random fluctuations of the sputtering yield. A careful experimental and theoretical study of the ion bombarding process by Andersen (22) shows, that a substantial transition layer is present with variations depending on energy and mass of the particles. Therefore a limit in depth resolution in the same range of thickness as the determined transition layer. The effect of ion bombardment on the LEED beam profiling technique has been studied by Schulze (23). Starting with a step free surface of germanium, the ion bombardment produced more steps than ever detected for oxidized silicon samples. The step density has been so high, that it was measurable only after a reduction due to a heat treatment. Even then an extraordinary high density has been found. It is therefore concluded that the transition layer as found during ion bombardment is not the same kind of information as found with the LEED technique.

Another important method for structural information is the transmission electron microscopy. A very thin cross section (10 nm) of an MOS structure is studied directly in the microscope. Blanc and coworkers (20) found a sharp and flat boarder line between Si and SiO₂ down to the resolution of 1 nm (corresponding to about 3 atomic layers). Krivanek and coworkers (21) demonstrated even with a resolution of 0.3 nm, that the transition is abrupt and that there are steps in distances of about 2 to 4 nm at the surface of silicon (which corresponds to widths of 6 to 12 atomic distances). Those results are in agreement with the results of this paper. Although TEM provides direct information on the presence of steps (even without removal of the oxide). This technique is not suited for routine work and precision determination of the step density due to its extreme effort in sample

preparation and quality in microscopy technique.

Indirectly the presence of steps have been concluded from the electron mobility in inversion layers of MOS structures at high inversion and low temperatures. Cheng and Sullivan (24) showed, that varying the oxidation parameters varies also the roughness.

The present results are compatible with all other reported results. On the other hand the present results provide information on the structure at the interface which are not available with any other technique.

VI Conclusion

LEED studies of the Si-SiO₂ interface after removal of the oxide have shown, that a roughness on an atomic scale is present which has been not detected with any other method. Quantitative evaluation of systematic experiments reveal the dependance of some oxidation parameters: Whereas wet atmosphere, short oxidation times and contaminations at the interface yield a high roughness, a fairly smooth surface is obtained for dry oxygen with step free samples to start with, especially when a posttreatment in non-oxidizing atmosphere is added. The results supplement the numerous investigations on the interface with a wide variety of methods. The presented results now enable a direct check of a correlation between electron mobility and step density which has been indirectly concluded from electrical measurements. Therefore the present results open a new way to understand and improve the technologically important interface Si-SiO₂.

Appendix

Main-program: HALED2

FORTRAN IV V01C-03A FRI 11-JAN-80 00:09:12 PAGE 001

```
      C      HAUPTPROGRAMM
      C      PROGRAMM ZUR BERECHNUNG DER RELATIVEN HALBWERTSWEITEN
0001      C      DIMENSION IBUF(2048)
      C      EICHUNG DES NORMALREFLEXABSTANDES:
0002      AM=150.0
0003      AB=3.326
0004      NEI=0
      C      ANFANGSDIALOG
0005      WRITE(7,49)
0006  49      FORMAT(1H,'NEU-EICHEN(0),EINGABE D. ALTEN EICHF.(1)')
0007      READ(7,41) KONST
0008  41      FORMAT(I2)
0009      IF(KONST.EQ.1) GOTO 98
0011      WRITE(7,19)
0012  19      FORMAT(1H,'1. EICHUNG DES NORMALREFLEXABSTANDES')
0013      WRITE(7,11)
0014  11      FORMAT(1H,'ANZAHL DER EICHMESSUNGEN = ?')
0015      READ(7,12)NEICH
0016  12      FORMAT(I4)
0017      IF(NEICH.EQ.0)NEICH=1
0019      YCL=0.0
0020      NEIC=0
0021  14      NEI=NEI+1
0022      WRITE(7,21)
0023  21      FORMAT(1H,'ELEKTROENENERGIE = ?')
0024      READ(7,22)MVOLT
0025  22      FORMAT(I4)
      C      ANFANGSDIALOG
0026      WRITE(7,10)
0027  10      FORMAT(1H,'MESSKURVENANZAHL = ?')
0028      READ(7,20) NMESS
0029  20      FORMAT(I4)
0030      IF(NMESS.EQ.0) NMESS=1
0032      IF(NMESS.GT.101) NMESS=1
0034      DO 30 I=1,2048
0035  30      IBUF(I)=0
      C      EINGABE DES MULTIPLIKATORS FUER INTEGRATIONSZEIT
      C      ZEIT=ITIME*80MSEC
0036      WRITE(7,40)
0037  40      FORMAT(1H,'80MSEC* ? =')
0038      READ(7,50) ITIME
0039  50      FORMAT(I3)
0040      IF(ITIME.EQ.0) ITIME=1
0042      IF(ITIME.GT.50) ITIME=1
      C      BEGINN DES MESSZYKLUS
0044      CALL TM109(NMESS,ITIME,IBUF)
      C      AUSGABE DES SPEICHERS
0045      DO 18 I=1,2048
0046  18      I2UF(I)=IBUF(I)/NMESS
0047      IF(FLOAT(NMESS)/2.0-FLOAT(NMESS/2).EQ.0.0) GOTO 99
0049      DO 99 I=1,2048,1
0050      IX=I*2
0051      IY=IBUF(I)*16
0052      CALL SUBR1(IX,IY)
```

FORTRAN IV

VOIC-03A

FRI 11-JAN-80 00:08:12

PAGE 002

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0053 99  CONTINUE
0054      CALL MAXIM(IBUF,10,2038,MAF,MAA)
0055      WRITE(7,3)MAF,MAA
0056 3    FORMAT(1H,'1.MAXIMUM: MAF = ',I4,' MAA = ',I4)
C      1.RELATIVES MINIMUM OBERHALB DES 1.MAXIMUM
0057      MAAX1=MAA
0058      N=MAAX1+200
0059 23   N=N+1
0060      M1=IBUF(N)
0061      M2=IBUF(N+1)
0062      IF(M1.GE.M2) GOTO 23
0064      M3=IBUF(N+2)
0065      IF(M1.GE.M3) GOTO 23
0067      IF(N+2.GE.2038) GOTO 24
0069      CALL MAXIM(IBUF,N+2,2038,MAF,MAA)
0070      MAF0=MAF
0071      MAA0=MAA
0072      N=N+2
0073      WRITE(7,9)
0074 9    FORMAT(1H,'ARG.OB.REL.MIN. FUN.OB.REL.MAX. ARG.OB.REL.MAX.')
0075      WRITE(7,4)N,MAF0,MAA0
0076 4    FORMAT(1H,'15,' ',15,' ',15,' ')
C      1.RELATIVES MINIMUM UNTERHALB DES 1. MAXIMUM
0077      N=MAAX1-200
0078 26   N=N-1
0079      M1=IBUF(N)
0080      M2=IBUF(N-1)
0081      IF(M1.GE.M2) GOTO 26
0083      M3=IBUF(N-2)
0084      IF(M1.GE.M3) GOTO 26
0086      IF(N-2.LE.10) GOTO 24
0088      CALL MAXIM(IBUF,10,N-2,MAF,MAA)
0089      MAFU=MAF
0090      MAAU=MAA
0091      N=N-2
0092      WRITE(7,39)
0093 39   FORMAT(1H,'ARG.UN.REL.MIN. FUN.UN.REL.MAX. ARG.UN.REL.MAX.')
0094      WRITE(7,5)N,MAFU,MAAU
0095 5    FORMAT(1H,'15,' ',15,' ',15,' ')
0096      MAAX2=MAAU
0097      IF(MAF0.GT.MAFU) MAAX2=MAA0
0099      NXY=IABS(MAAX1-MAAX2)
0100      WRITE(7,6)NXY
0101 6    FORMAT(1H,'ABSTAND DER REFLEXE : ',I5)
0102      XY=FLOAT(NXY)
0103      VOLT=FLOAT(MVOLT)
0104      NEIC=NEIC+1
0105      YZ=XY*AB*SQRT(VOLT/AM)
0106      WRITE(7,31) YZ
0107 31   FORMAT(1H,'EICHUNG : Y(CL) = ',F8.2)
0108      YCL=YCL+YZ
0109      GOTO 13
0110 24   WRITE(7,33)
0111 33   FORMAT(1H,'REFLEXE ZU NAHE AN DEN FELDGERENZEN')

```


FORTRAN IV

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```

0112 13  CONTINUE
0113      IF(NEI.NE.NEICH) GOTO 14
        C  MITTELUNG
0115      EIC=FLOAT(NEIC)
0116      YCLM=YCL/EIC
0117      WRITE(7,29)YCLM
0118 29  FORMAT(1H , 'EICHUNG:MITTELW.:Y(CLM) = ',F8.2)
        C  NORMALREFLEXABSTAND=YCLA/SQRT(VOLT)
0119      YCLA=YCLM*SQRT(AM)/AB
0120      WRITE(7,59)YCLA
0121 59  FORMAT(1H , 'EICHKONSTANTE : YCLA = ',F8.2)
0122      GOTO 77
        C  EINGABE DER ALTEN EICHKONSTANTE , DA DER RECHNER
        C  AUSGESTIEGEN IST
0123 88  WRITE(7,69)
0124 69  FORMAT(1H , 'EINGABE DER ALTEN EICHKONSTANTEN')
0125      READ(7,81) YCLA1
0126 51  FORMAT(F8.2)
0127      YCLA=YCLA1
0128 77  CONTINUE
        C  PROBELAUF: PROGRAMMTEIL ZUR BERECHNUNG DER
        C  POSITION DES AUSZUMESSENDEN REFLEXES
        C  MESSPROGRAMM FUER TRANSIENTENREKORDER
        C  ANFANGSDIALOG
0129      WRITE(7,111)
0130 111  FORMAT(1H , 'BEST. DES MESSINTERVALLES: MESSKURVENANZAHL = ?')
0131      READ(7,120)NMESS
0132 120  FORMAT(I4)
0133      IF(NMESS.EQ.0) NMESS=1
0135      DO 130 I=1,2048
0136 130  IBUF(I)=0
        C  EINGABE DES MULTIPLIKATORS FUER INTEGRATIONSZEIT
        C  ZEIT=ITIME*80MSEC
0137      WRITE(7,140)
0138 140  FORMAT(1H , '80MSEC* ? =')
0139      READ(7,150)ITIME
0140 150  FORMAT(I3)
0141      IF(ITIME.EQ.0) ITIME=1
        C  BEGINN DES MESSZYKLUS
0143      CALL TM109(NMESS,ITIME,IBUF)
        C  NORMIERUNG DES SPEICHERS
0144      DO 168 I=1,2048
0145 168  IBUF(I)=IBUF(I)/NMESS
0146      CALL MAXIM(IBUF,10,2038,MAF,MAA)
0147      WRITE(7,102) MAF,MAA
0148 102  FORMAT(1H , 'MAX. DES REFL. : MAF = ',I5, ' MAA = ',I5)
0149      NOG=MAA+200
0150      NUG=MAA-200
0151      WRITE(7,103) NOG,NUG
0152 103  FORMAT(1H , 'NOG = ',I5, ' NUG = ',I5)
        C  PROGRAMMTEIL ZUR BERECHNUNG DER
        C  RELATIVEN HALBWERTSBREITEN--MESSEN--
0153      WRITE(7,211)
0154 211  FORMAT(1H , 'MESSEN')

```

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```

0155 222 CONTINUE
0156 WRITE(7,251)
0157 251 FORMAT(1H,'ELEKTRONENENERGIE(M) = ?')
0158 READ(7,232)MVOL
0159 232 FORMAT(I4)
0160 FOCUS=0.7619*FLOAT(MVOL)-21.7143
0161 WRITE(7,288) FOCUS
0162 288 FORMAT(1H,'FOCUS = ',F4.1,'VOLT')
C MESSPROGRAMM FUR TRANSIENTENREKORDER
C ANFANGSDIALOG
0163 WRITE(7,291)
0164 291 FORMAT(1H,'MESSKURVENANZAHL = ?')
0165 READ(7,220)NMESS
0166 220 FORMAT(I4)
0167 IF(NMESS.EQ.0) NMESS=1
0169 IF(NMESS.GT.101) NMESS=1
0171 DO 230 I=1,2048
0172 230 IBUF(I)=0
C EINGABE DES MULTIPLIKATORS FUER INTEGRATIONSZEIT
C ZEIT=ITIME*80MSEC
0173 WRITE(7,240)
0174 240 FORMAT(1H,'80MSEC* = ?')
0175 READ(7,250) ITIME
0176 250 FORMAT(I3)
0177 IF(ITIME.EQ.0) ITIME=1
0179 IF(ITIME.GT.50) ITIME=1
C BEGINN DES MESSZYKLUS
0181 CALL TM109(NMESS,ITIME,IBUF)
C NORMIERUNG DES SPEICHERS
0182 DO 268 I=1,2048
0183 268 IBUF(I)=IBUF(I)/NMESS
C BESTIMMUNG DES MAXIMUMS IM INTERVALL
0184 CALL MAXIM(IBUF,NUG,NOG,MAF,MAA)
0185 MAFY=MAF
0186 MAAX=MAA
0187 WRITE(7,201)MAF,MAA
0188 201 FORMAT(1H,'MAF = ',I5,' MAA = ',I5)
C BESTIMMUNG DES ABSOLUTEN MINIMUMS
0189 NUG1=MAAX-500
0190 NOG1=MAAX+500
0191 CALL MINIM(IBUF,NUG1,NOG1,MIF,MIA)
0192 MIFY=MIF
0193 MIAX=MIA
0194 WRITE(7,202)MIF,MIA
0195 202 FORMAT(1H,'MIF = ',I5,' MIA = ',I5)
C BERECHNUNG DES UNTERGRUNDES
0196 ADELTA=FLOAT(MAFY-MIFY)
0197 A6PR=6.0*ADELTA/100.0
C ZAEHLUNG ALLER FUNKTIONSWERTE, DIE IM FENSTER
C MINIMUM +6% LIEGEN
0198 A6GR=FLOAT(MIFY)+A6PR
0199 SUM=0.0
0200 M=0
0201 DO 243 I=NUG1,NOG1

```

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```

0202      AN=FLOAT(IBUF(I))
0203      IF(AN.GT.0.66R) GOTO 243
0205      M=M+1
0206      SUM=SUM+AN
0207 243   CONTINUE
0208      UNT=SUM/FLOAT(M)
0209      WRITE(7,203)UNT
0210 203   FORMAT(1H,'UNTERGRUND = ',F8.2)
      C  MAXIMUM-UNTERGRUND-DIFF: DIF2-DIFF/2
0211      DIF2=(FLOAT(MAFY)-UNT)/2.0
0212      DIF2=DIF2+UNT
      C  BESTIMMUNG DER GEMESSENEN HALBWERTSBREITE
      C  OBERER WERT
0213      N=MAAX
0214 244   N=N+1
0215      SX=FLOAT(IBUF(N))
0216      IF(SX-DIF2.GT.0.0) GOTO 244
0218      S1=FLOAT(IBUF(N-1))-DIF2
0219      S2=FLOAT(IBUF(N))-DIF2
0220      S2=ABS(S2)
0221      IF(S1.GT.S2) NO=N
0223      NO=N-1
      C  UNTERER WERT
0224      N=MAAX
0225 255   N=N-1
0226      SX=FLOAT(IBUF(N))
0227      IF(SX-DIF2.GT.0.0) GOTO 255
0229      S1=FLOAT(IBUF(N+1))-DIF2
0230      S2=FLOAT(IBUF(N))-DIF2
0231      S2=ABS(S2)
0232      IF(S1.GT.S2) NU=N
0234      NU=N+1
0235      WRITE(7,204) NU,NO
0236 204   FORMAT(1H,'NU = ',I5,' NO = ',I5)
      C  BESTIMMUNG DER GEMESSENEN HALBWERTSBREITE
0237      AHR=FLOAT(NO-NU)
0238      VOL=FLOAT(MVOL)
      C  BESTIMMUNG DER RELATIVEN HALBWERTSBREITE
0239      RHBR=SQRT(VOL)*AHR*100.0/YCLA
0240      WRITE(7,205)MVOL,RHBR
0241 205   FORMAT(1H,'ENERGIE = ',I4,' EV REL.HALWBW. = ',F5.2,' %')
0242      IF(FLOAT(NMESS)/2.0-FLOAT(NMESS/2).EQ.0.0) GOTO 222
      C  AUSGABE DES SPEICHERS
0244      DO 262 I=1,2048,1
0245      IX=I*2
0246      IY=IBUF(I)*16
0247      CALL SUBR1(IX,IY)
0248 262   CONTINUE
0249      GOTO 222
0250      STOP
0251      END

```

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```
0001      SUBROUTINE MAXIM(IBUF,NUN,NOB,MAF,MAA)
0002      DIMENSION IBUF(2048)
0003      MAF=0
0004      DO 1 I=NUN,NOB
0005      MAF1=IBUF(I)
0006      IF(MAF1.LT.MAF) GOTO 1
0008      MAF=MAF1
0009      MAA=I
0010    1    CONTINUE
0011      RETURN
0012      END
```

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```
0001      SUBROUTINE MINIM(IBUF,NUN,NOB,MIF,MIA)
0002      DIMENSION IBUF(2048)
0003      MIF=256
0004      DO 2 I=NUN,NOB
0005      MIF1=IBUF(I)
0006      IF(MIF1.GT.MIF) GOTO 2
0008      MIF=MIF1
0009      MIA=I
0010    2    CONTINUE
0011      RETURN
0012      END
```

Macro Subroutine: TM109

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```

1          .TITLE   TM109
2          .GLOBL   TM109
3          .MCALL   ..V2...REGDEF
4          167760    STATUS=167760
5          167762    OUTP=STATUS+2
6          167764    INP=STATUS+4
7 000000    ..V2..
8 000000    .REGDEF
9 000000    ADRS:    .BLKW   1          ;ADRESSE VON IBUF(1)
10 00002    ITIME:   .BLKW   1          ;INTEGRATIONSZEIT
11 00004 005725 TM109: TST      (R5)+    ;DUMMY
12 00006 013504    MOV      0(R5)+,R4      ;R4 ENTHAELT MESSANZAHL
13 00010 013567    MOV      0(R5)+,ITIME    ;ITIME ENTHAELT INTEGRATIONSZEIT
14 00014 010567    MOV      R5,ADRS ;ANFANGSADRESSE VON IBUF
15 00020 012767 START: MOV      #000002,OUTP    ;RESET HIGH, TRIGGER LOW
16 00026 042767    BIC      #000002,OUTP    ;RESET RECORDER BIT 1
17 00034 052767    BIS      #000002,OUTP
18 00042 000240    NOP
19          ;ABFRAGE ERSTES BILD ?
20 00044 032767 1$:    BIT      #001000,INP    ;BIT 9 LESEN
21 00052 001774    BEQ      1$
22 00054 052767    BIS      #000004,OUTP    ;START INTEGRATION; BIT 2
23 00062 016701    MOV      ITIME,R1
24 00066 012700 2$:    MOV      #-8730.,R0      ;WARTESCHLEIFE
25 00072 005200 3$:    INC      R0
26 00074 002776    BLT      3$
27 00076 005301    DEC      R1
28 00100 001372    BNE      2$
29          ;ABFRAGE VIERTES BILD ?
30 00102 032767 4$:    BIT      #002000,INP    ;BIT 10 LESEN
31 00110 001774    BEQ      4$
32 00112 042767    BIC      #000004,OUTP    ;STOP INTEGRATION BIT 2
33          ;ABFRAGE ERSTES BILD ?
34 00120 032767 5$:    BIT      #001000,INP    ;BIT 9 LESEN
35 00126 001774    BEQ      5$
36 00130 052767    BIS      #000001,OUTP    ;TRIGGER DES RECORDERS

```

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```

167762'
37 00136 042767      BIC      #000001,OUTP
      000001
      167762'
38 00144 000240      NOP
39      ;WARTESCHLEIFE BIS AUFNAHME BEENDET IST
40      ;ABFRAGE BIT 8 DES DIGITALEN EINGANGES
41 00146 032767 10$:  BIT      #000400,INP      ;BIT 8 LESEN
      000400
      167764'
42 00154 001374      BNE      10$
43      ;LESEN DES SPEICHERS
44 00156 000240      NOP
45 00160 012700      MOV      #-2048.,R0      ;ZAEHLER SETZEN
      174000
46 00164 016701      MOV      ADRS,R1
      177610
47 00170 116703 LOOP:  MOV     INP,R3
      167764'
48 00174 042703      BIC      #177400,R3
      177400
49 00200 060321      ADD      R3,(R1)+
50 00202 005200      INC      R0
51 00204 001371      BNE      LOOP
52 00206 005304      DEC      R4      ;NMESS HERABSETZEN
53 00210 001303      BNE      START
54 00212 000207      RTS      PC      ;BACK TO PROGRAMM
55      000004'      .END      TM109

```

TM109 RT-11 MACRO VM02-12 11-JAN-80 02:59:44 PAGE.14
 SYMBOL TABLE

ADRS	000000R	INP	= 167764	ITIME	000002R
LOOP	000170R	OUTP	= 167762	PC	=%000007
R0	=%000000	R1	=%000001	R2	=%000002
R3	=%000003	R4	=%000004	R5	=%000005
SP	=%000006	START	000020R	STATUS	= 167760
TM109	000004RG	...V2	= 000001		
. ABS.	000000				
	000214	000			
		001			

ERRORS DETECTED: 0
 FREE CORE: 17658. WORDS

TM109,TT:=TM109

ERRORS DETECTED: 0
 FREE CORE: 17658. WORDS

*

Macro Subroutine: SUBR1

.RU DX1:MACRO

*SUBR1,TT:=SUBR1

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```

1          .TITLE  SUBR1
2          .GLOBL  SUBR1
3          .MCALL  ..V2...REGDEF
4          170420  REGISX=170420
5          170422  REGISY=170422
6 000000    ..V2..
7 000000    .REGDEF
8 000000 005725 SUBR1: TST      (R5)+
9 000002 013567      MOV      @ (R5)+,REGISX
          170420'
10 00006 017567      MOV      @ (R5),REGISY
          000000
          170422'
11          ;SPEICHERINHALT IST IM AUSGABEREGISTER
12          ;WAIT A LITTLE
13 00014 012700      MOV      #-1000.,R0
          176030
14 00020 005200 1$:   INC      R0
15 00022 003776      BLE      1$
16 00024 000207      RTS      PC      ;ZURUECK ZUM HAUPTPROGRAMM
17          000000'    .END      SUBR1

```

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SYMBOL TABLE

PC	=%000007	REGISX=	170420	REGISY=	170422
R0	=%000000	R1	=%000001	R2	=%000002
R3	=%000003	R4	=%000004	R5	=%000005
SP	=%000006	SUBR1	000000RG	...V2 =	000001

. ABS. 000000 000
000026 001

ERRORS DETECTED: 0

FREE CORE: 17698. WORDS

SUBR1,TT:=SUBR1

ERRORS DETECTED: 0

FREE CORE: 17698. WORDS

*

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Figure Captions

- Fig. 1: Schematic configuration of LEED and closed TV-system
- Fig. 2: Examples for oxide thickness determination with Auger electron spectroscopy
- a) Spectrum of a clean silicon surface
 - b) Si with a layer of about 2.7 nm SiO_2
 - c) Si with a layer of SiO_2 larger than 12 nm
- Fig. 3: a) Auger spectrum of a crystal directly after removal of the oxide and transfer in the UHV
- b) Auger spectrum of the sample holder after several experimental cycles
- Fig. 4: Relative half width of the 00-spot versus energy for a well annealed probe (7x7) structure, etched in HF and transferred back to UHV. The upper curve is calculated for an edge atom density of 3.8% and is the best for the experimental points (crosses). The lower curve gives the instrumental limit. S (an multiples of 2π) indicates the energies at which in-phase scattering happens.
- Fig. 5: Relative half width versus energy for the same sample after the following treatments:
- a) Oxidation 30 sec at 1000°C in dry oxygen (crosses xxx) upper curve calculated for a half width of about 12%
 - b) Annealing at 1000°C (7x7 structure), yielding instrumental limit (lower curve, circles 000).
 - c) Annealing at 1000°C , transfer to atmosphere, etching in HF, transfer back to UHV, showing a residual roughness (center curve: crosses ++).

- Fig. 6:** Relative half width versus energy for the same sample after the following treatments:
- a) Oxidation 16 h at 1000° C in dry oxygen, etching in HF (crosses: xxx).
 - b) Second etching in HF without annealing (crosses: +++).
 - c) Third etching in HF without annealing (crossed circles: ØØØ).
 - d) Etching in HF after annealing at 1000° C.
Upper curve is calculated for a half width of about 11.7%, center curve for 3%.

- Fig. 7:** Corrected relative half width and step atom density versus position on the crystal for a sample with a wide scatter of oxide thickness (upper curve) and a sample with a light oxidation (overnight at room atmosphere, lower curve).

- Fig. 8:** Corrected relative half width and step atom density versus various oxidation pre- (1. - 5.) and posttreatment (6, 7) procedures

- a) with dipping into Methanol before etching in HF (crosses: xxx)
- b) without dipping into Methanol before etching in HF (circles: 000)

Oxidation parameters: 2 hours at 1000° C in dry oxygen

- Fig. 9:** Corrected relative half width and step atom density versus oxide thickness for two samples (crosses: x and circles: 0), which were well annealed in UHV (7x7 structure) before oxidation at 800° C in dry oxygen.
- Sample W1 (pretreatment: annealing in UHV), and sample W2 (pretreatment: oxidized and etched in HF) were oxidized in wet atmosphere.

Fig. 10: Corrected relative half width and step atom density versus oxide thickness for samples (crosses: xx) oxidized at 1000°C in dry atmosphere. Sample W3 has been oxidized in wet oxygen.

Fig. 11: Corrected relative half width and step atom density versus treatment before oxidation (2 h at 1000°C in dry oxygen)

1. Factory polished (by Wacker Chemitronic, Burghausen) (crosses: xxx).
2. Chemically etched with CP6 (circles: 000).
3. Oxidized at 1000°C and oxide removed by HF (crossed circles: @@@)
4. Step free due to annealing in UHV (crossed circles: ØØØ)

1' and 3' indicates a second experimental run with the same treatment as in 1. und 3.

Fig. 12: Corrected relative half width and step atom density versus treatment before and after oxidation (2 hours at 1000°C in dry oxygen)

1. Factory polished
- 1' With additional annealing (5 hours in N_2 at 750°C)
3. Oxidized at 1000°C and oxide removed by HF before oxidation
- 3' With additional annealing (5 hours in N_2 at 750°C)

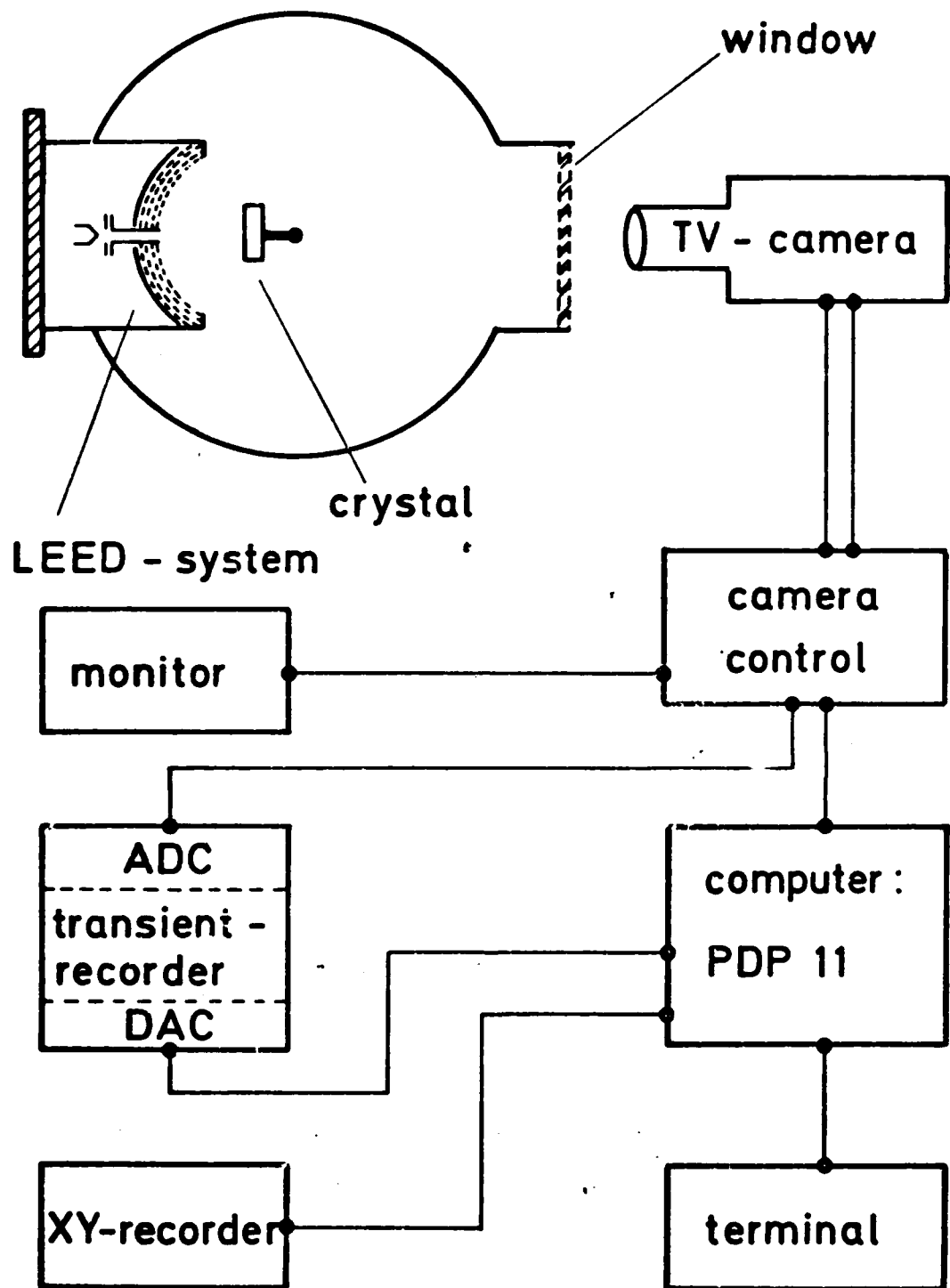


Fig. 1

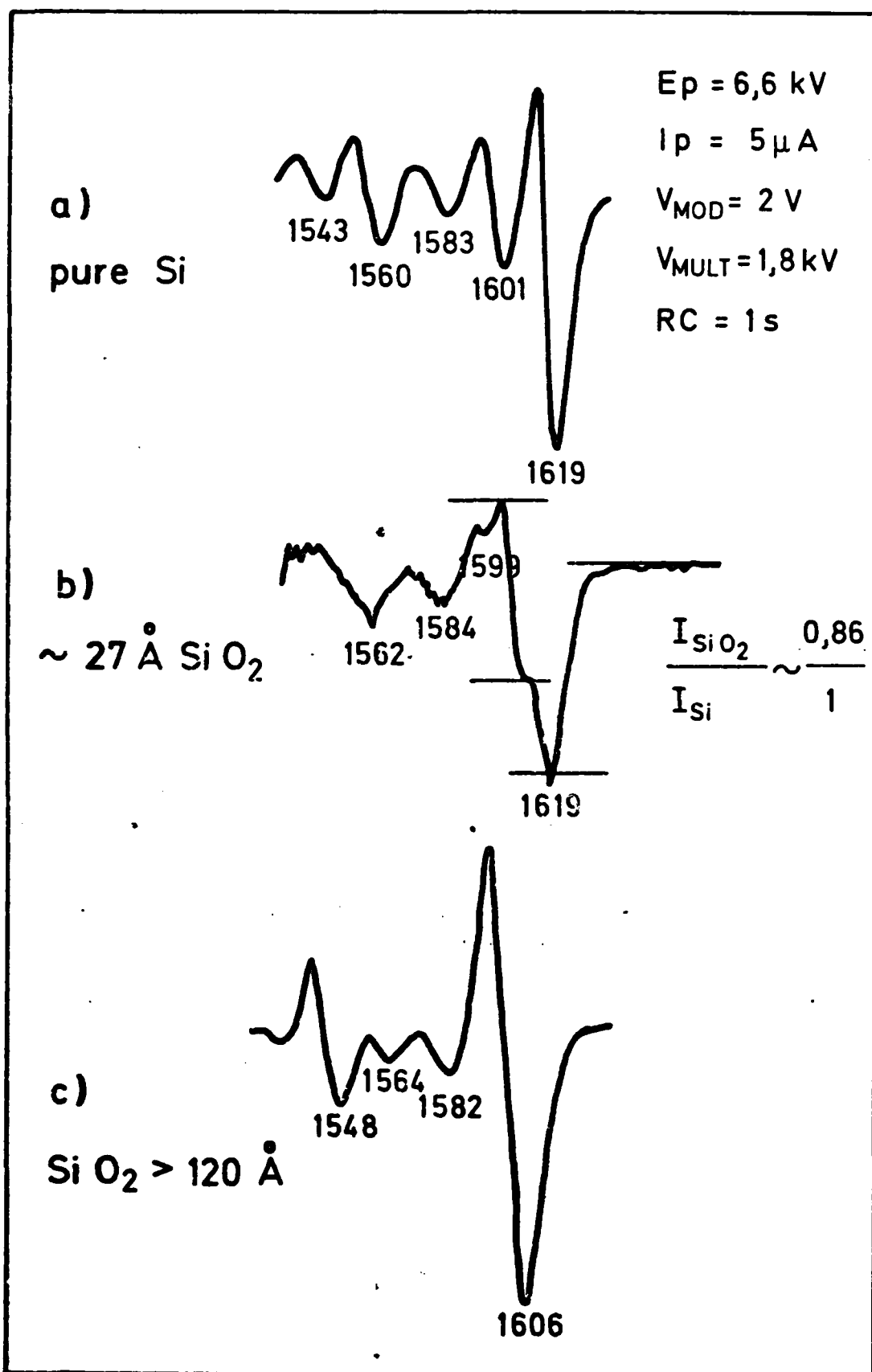


Fig. 2

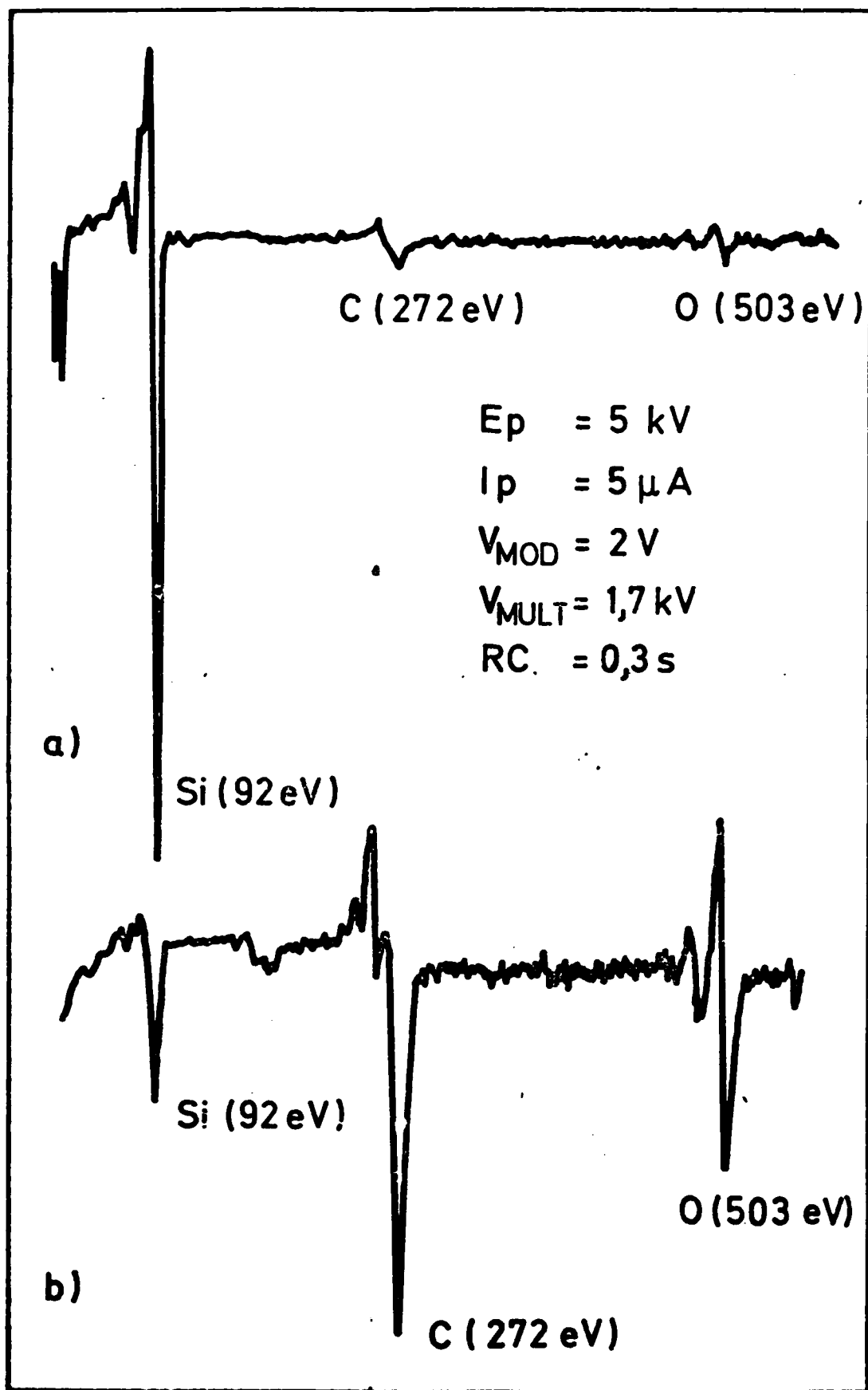


Fig. 3

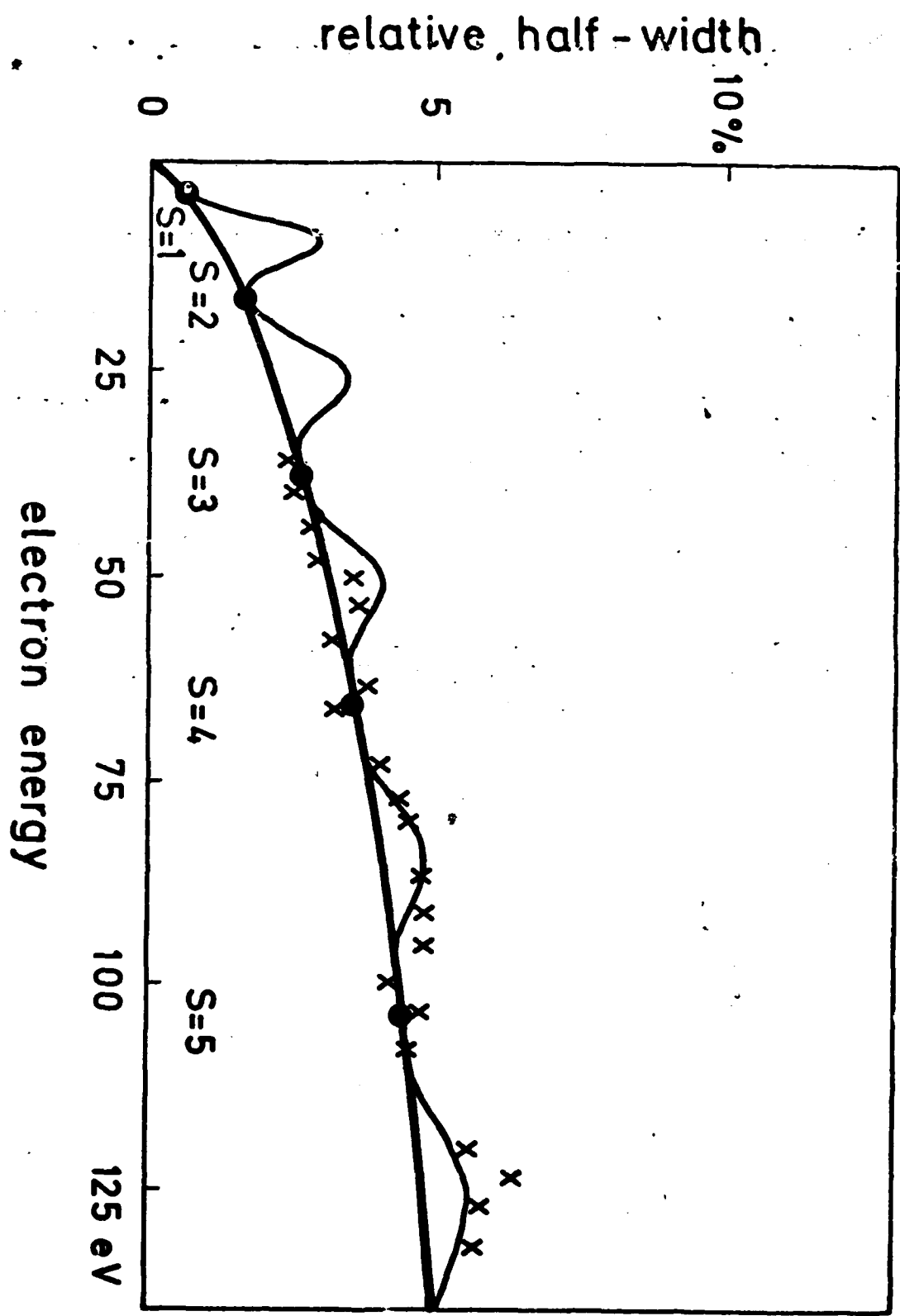


Fig. 4

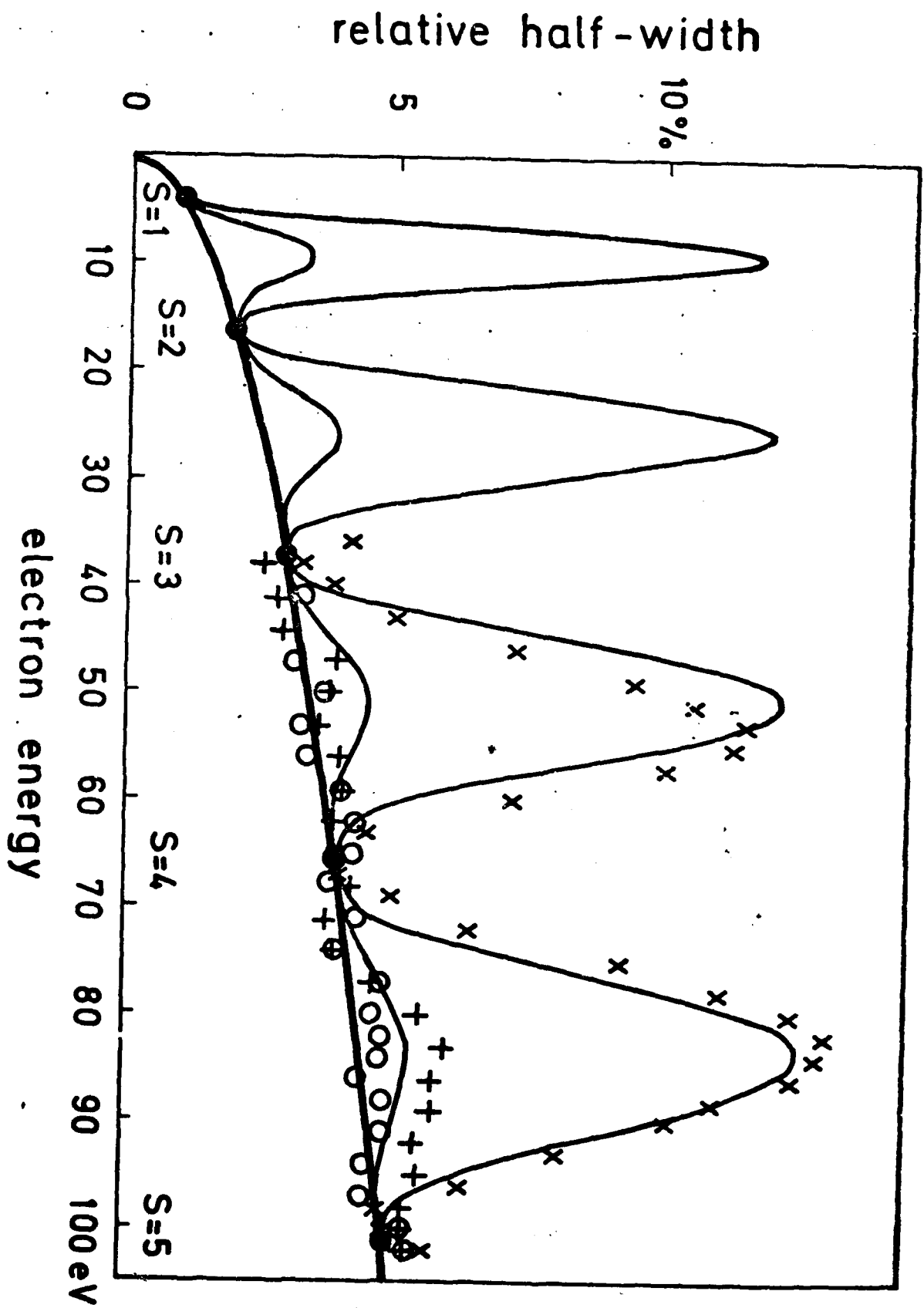


Fig. 5

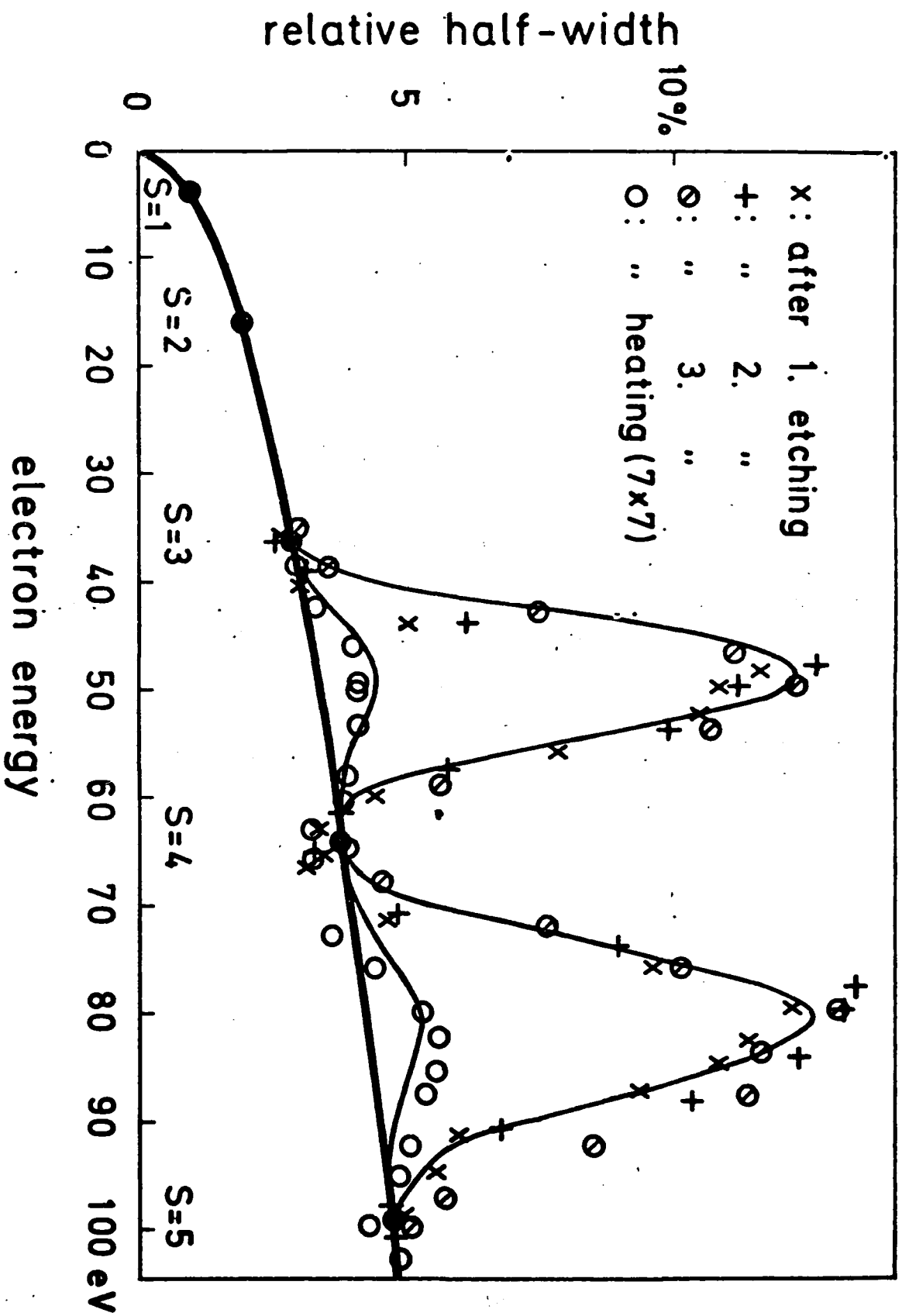
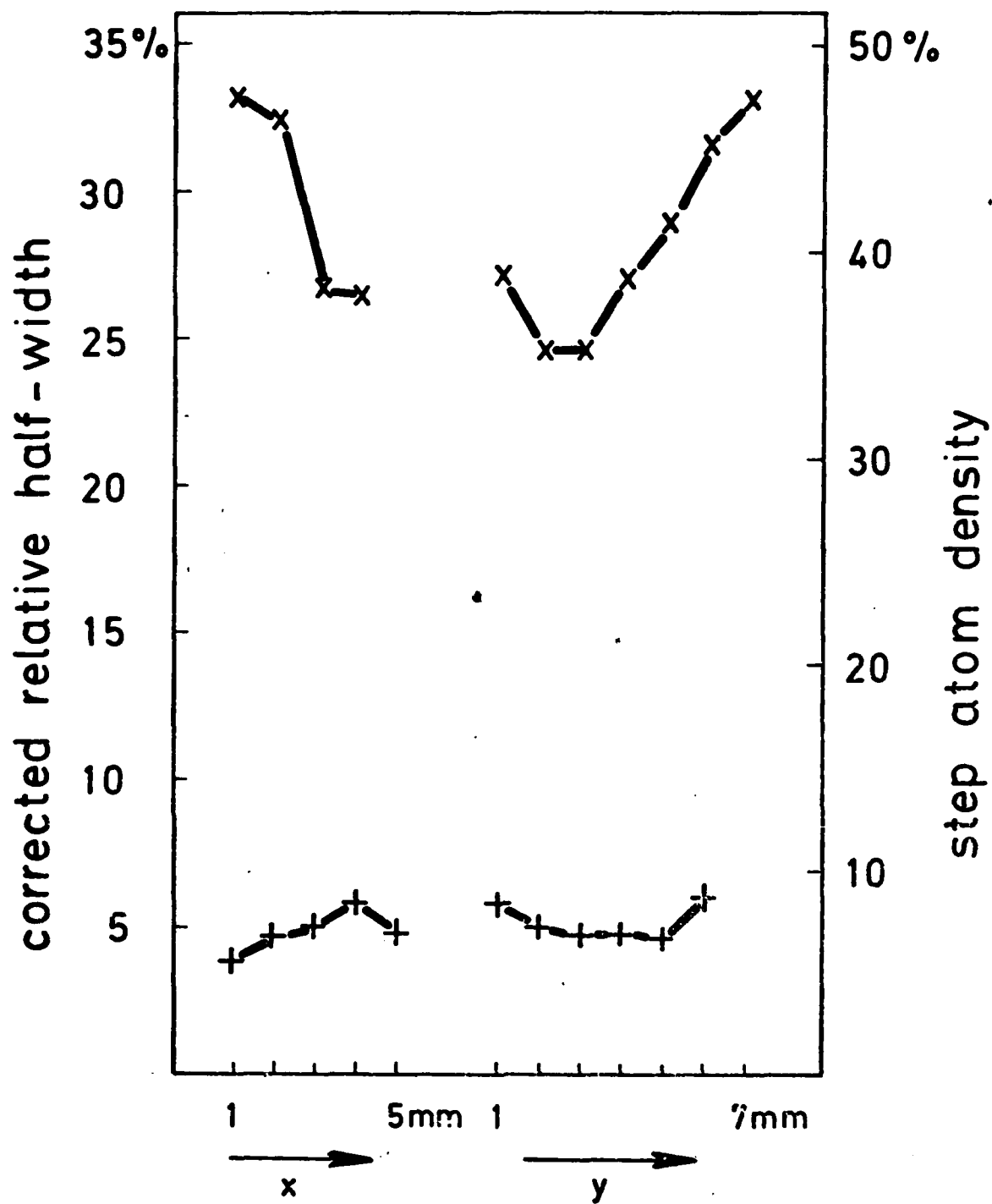


Fig. 6



position on crystal :

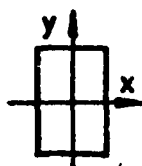


Fig. 7

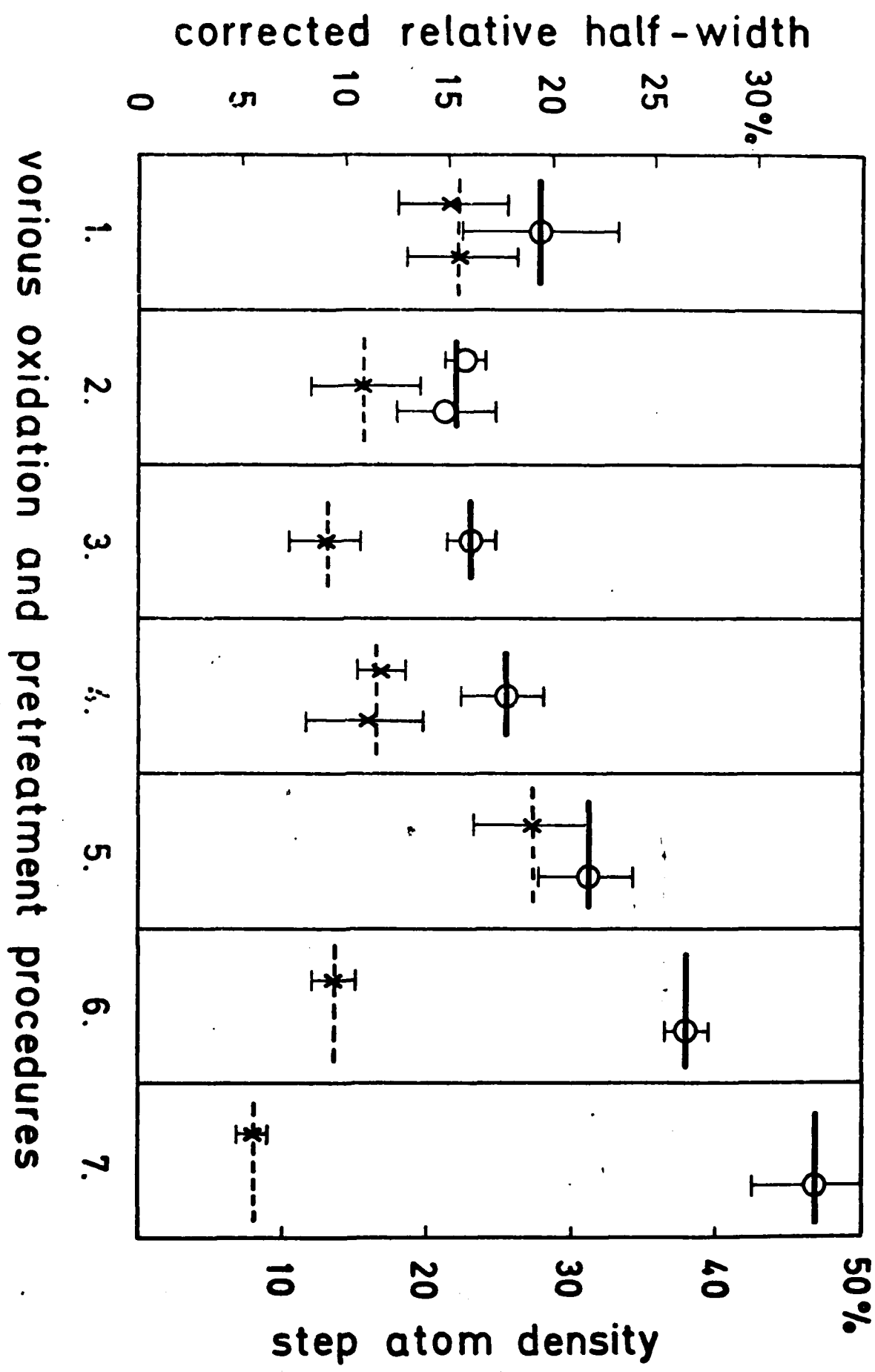


Fig. 8

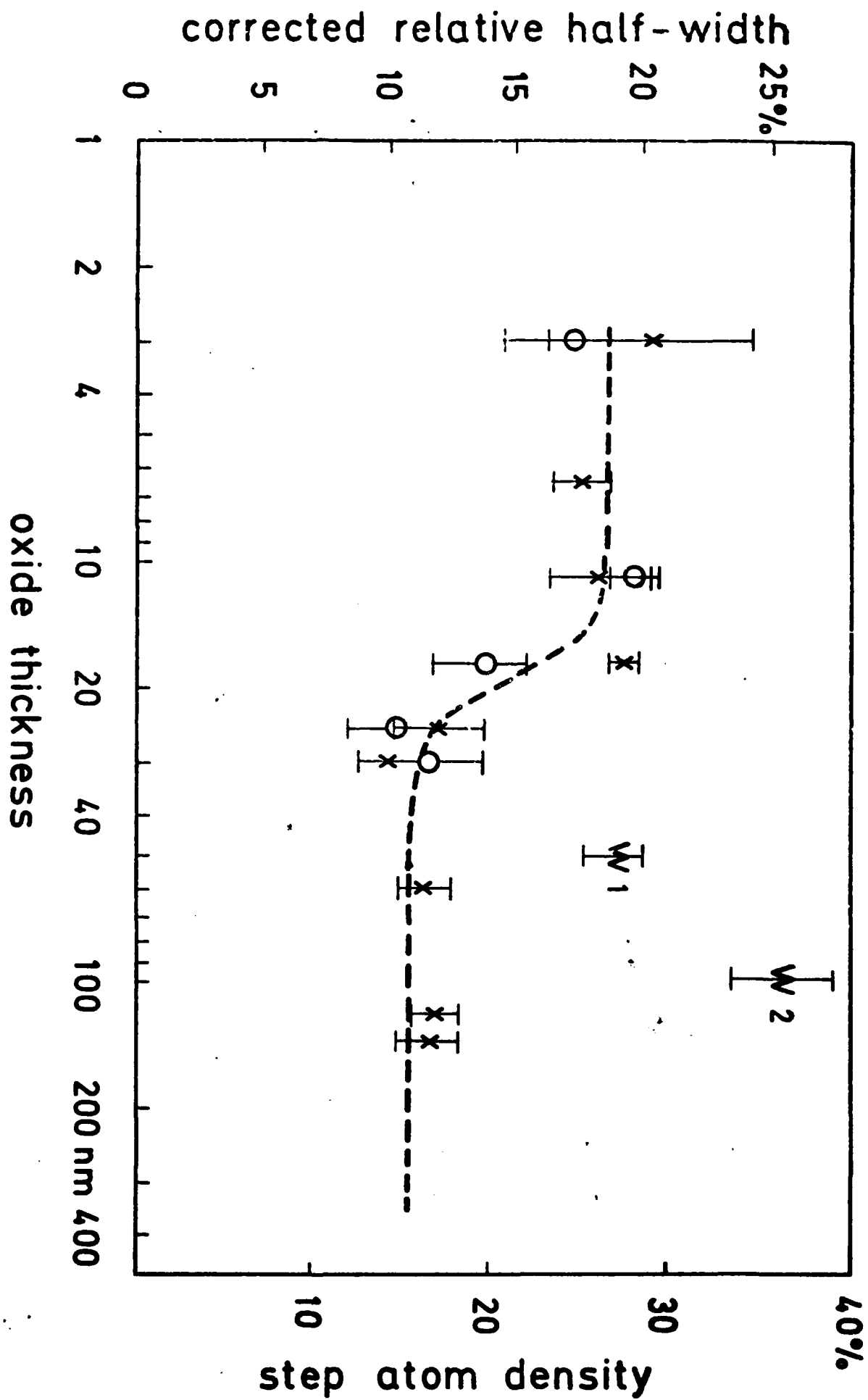


Fig. 9

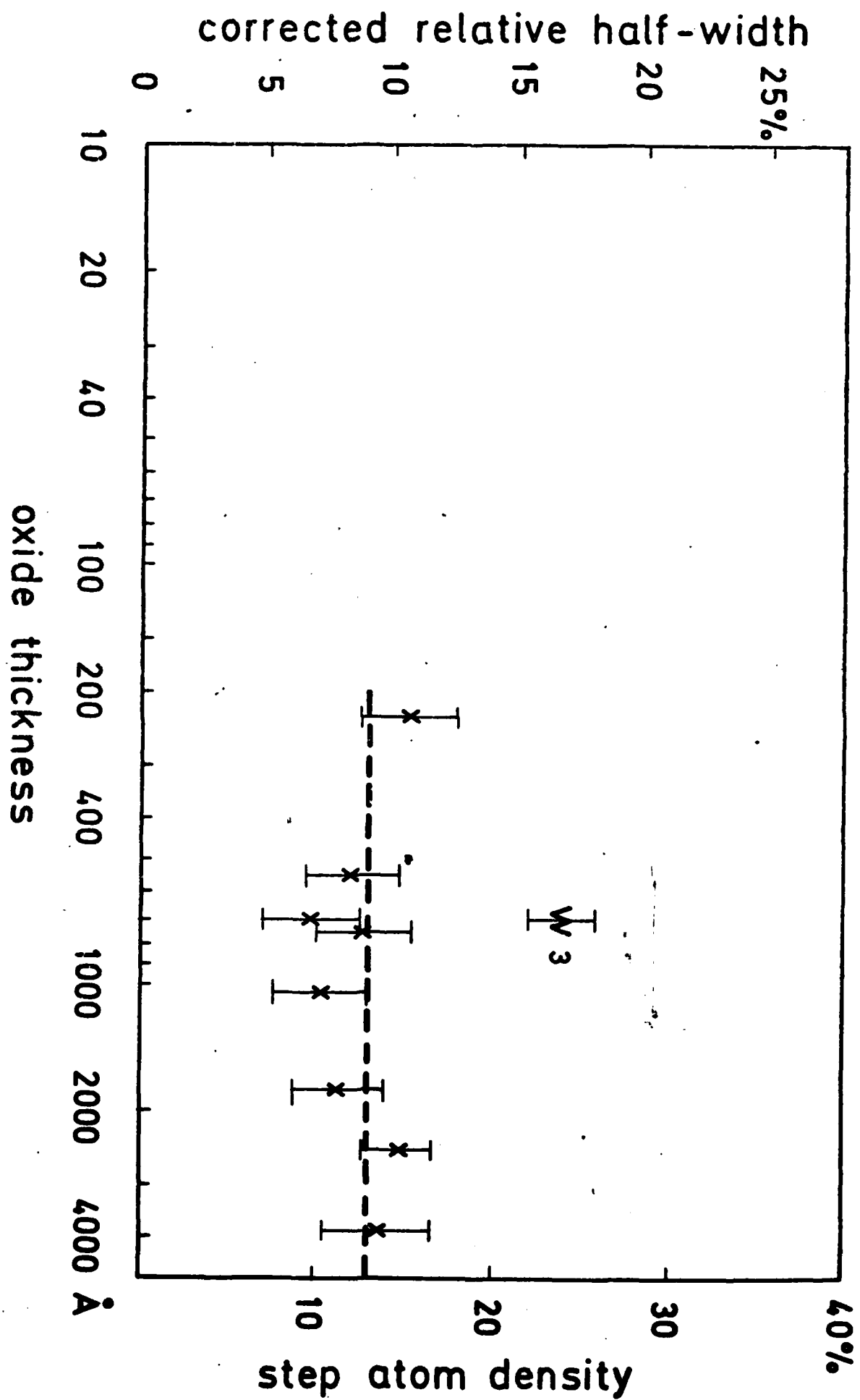


Fig. 10

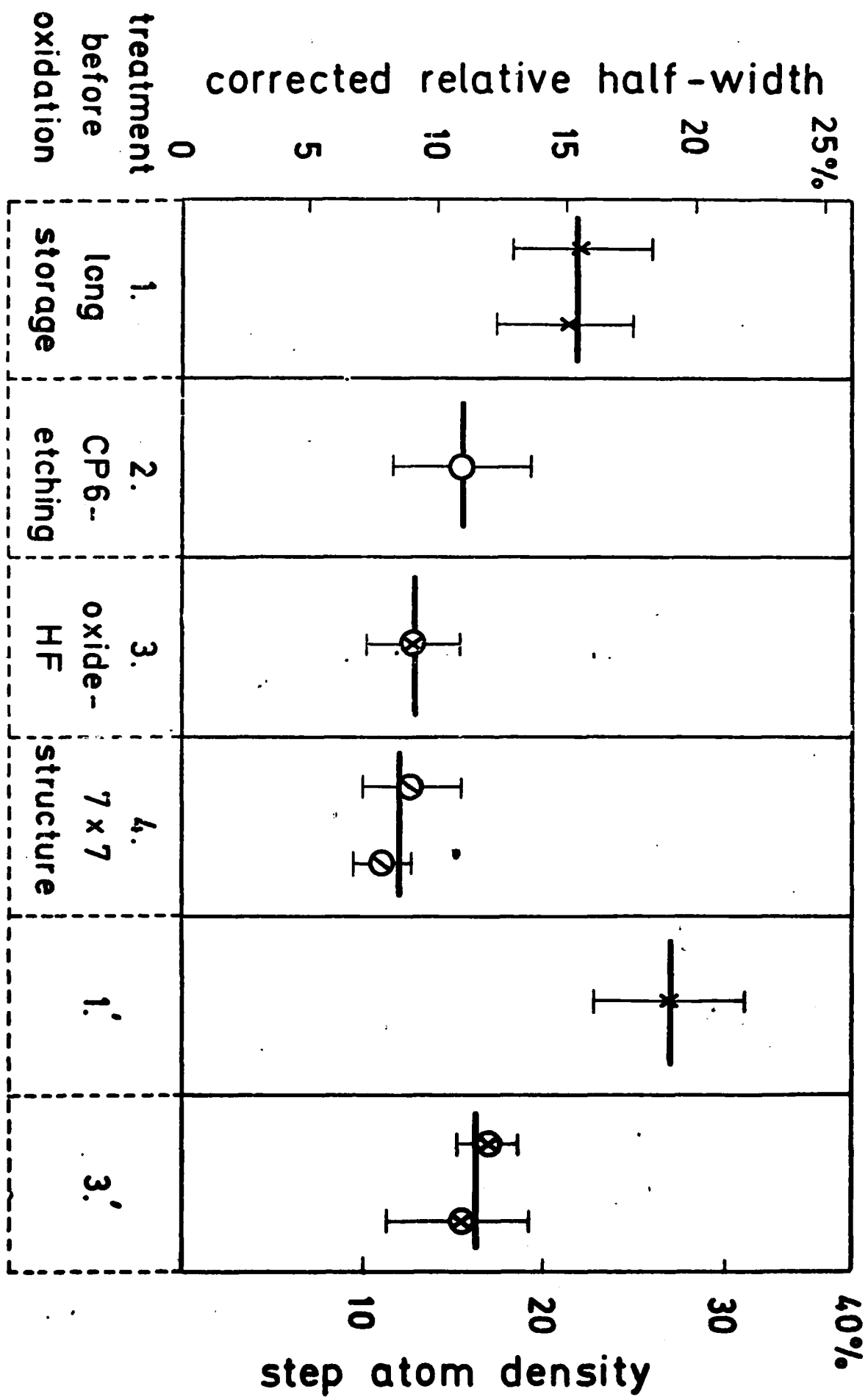


Fig. 11

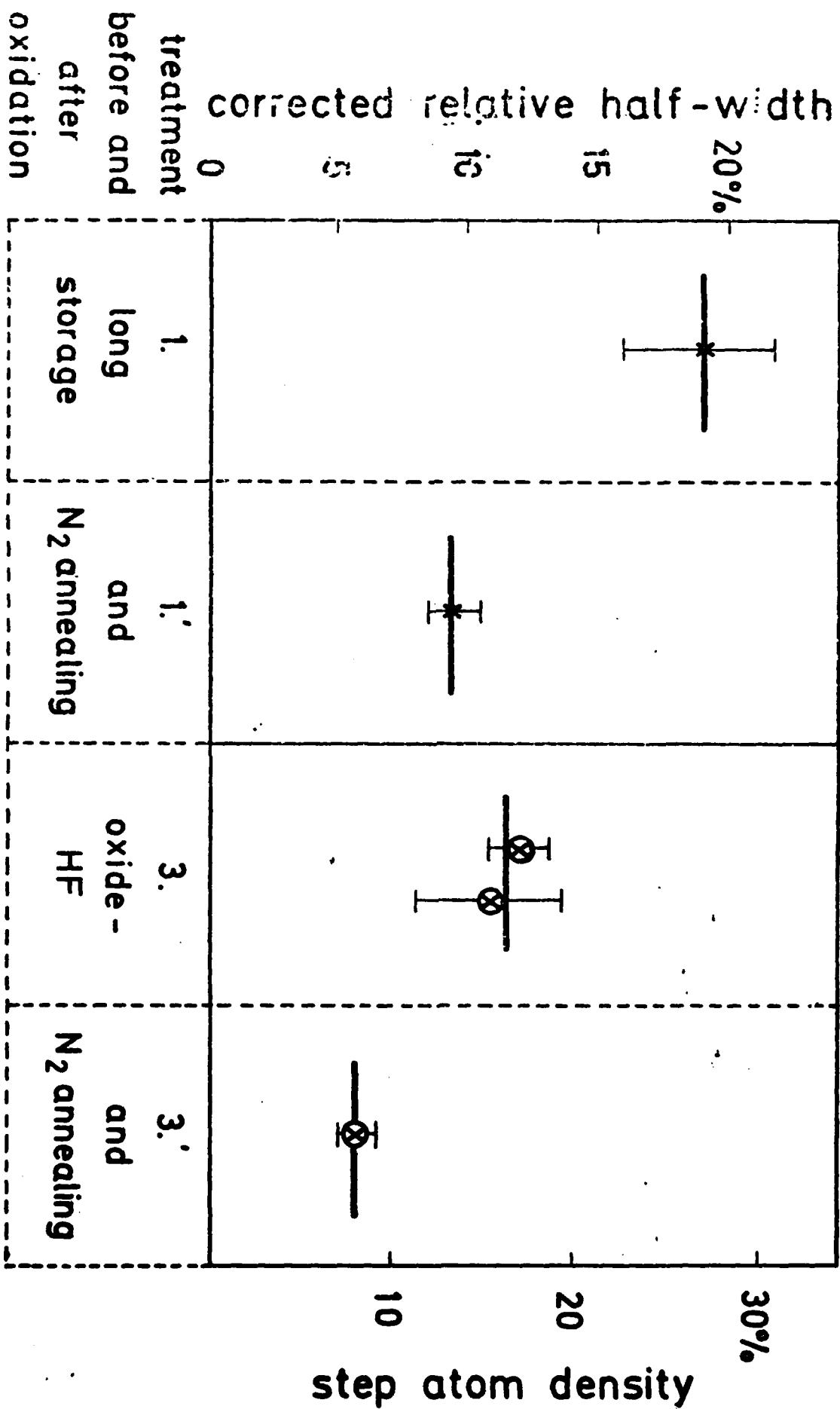


Fig.12

MED
8